



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 2775

#### Sulfur in Foundry Coke

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of test methods and for the calibration of instruments used to determine sulfur in foundry (metallurgical) coke. Each unit of SRM 2775 consists of 50 g of foundry coke that was ground to pass a 60 mesh (250  $\mu\text{m}$ ) sieve, homogenized, and bottled under an argon atmosphere.

Table 1. Certified Value (Mass Fraction)

|        |                         |
|--------|-------------------------|
| Sulfur | 0.5816 % $\pm$ 0.0051 % |
|--------|-------------------------|

**Certified Value:** A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified value is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS). The certified value for sulfur is reported in Table 1 as a mass fraction [1] on a dry basis (see “Instructions for Drying”). The uncertainty in the certified value is calculated as,  $U = ku_c$ , where  $u_c$  is the *combined standard uncertainty* calculated according to the NIST ISO Guide [2] and  $k$  is a coverage factor. The value of  $u_c$  represents, at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. The expanded uncertainty,  $U$  is based on a 95 % prediction interval. The coverage factor,  $k = 2.78$ , is the value from the Student’s  $t$ -distribution corresponding to four degrees of freedom and 95 % confidence.

**Reference Values:** Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty. Reference values for ash [3,4], volatile matter [4,5], carbon [7] hydrogen [7] and nitrogen [7] are reported in Table 2 as mass fractions [1] on a dry basis (see “Instructions for Drying”). The percent dry fixed carbon for SRM 2775 can be calculated by subtracting the sum of the dry ash and dry volatile matter from 100 % [6].

**Expiration of Certification:** This certification is valid until **31 December 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Use”). However, the certification will be nullified if the SRM is contaminated or modified.

The overall direction and coordination of the technical measurements leading to certification were performed by J.R. DeVoe, W.R. Kelly, P.A. Pella, R.D. Vocke, Jr., and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Certification analyses by ID-TIMS were performed by W.R. Kelly and R.D. Vocke of the NIST Analytical Chemistry Division. The X-ray fluorescence homogeneity analysis was performed by A.F. Marlow and P.A. Pella of the NIST Analytical Chemistry Division.

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*See Certificate Revision History on Last Page*

Statistical consultation was provided by K.R. Eberhardt, R.C. Hagwood, and D.D. Leber of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The foundry (metallurgical) coke for this SRM was donated by ABC COKE<sup>1</sup>, Birmingham, AL.

## NOTICE AND WARNINGS TO USERS

**Stability:** This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

## INSTRUCTIONS FOR USE

To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample mass of 150 mg should be used and the sample should be dried according to the “Instructions for Drying”. To relate analytical determinations to ash and volatile matter reference values, a nominal sample weight of 1 g should be used. To relate analytical determinations to carbon, hydrogen, and nitrogen reference values, a minimum sample mass of 90 mg should be used. When not in use, the SRM must be stored in an air conditioned or similar cool and dry environment away from sunlight and fumes.

**Instructions for Drying:** In order for users to directly relate their measurements to the certified value, loss on drying corrections should be measured and applied at the time of the analysis. The moisture correction for sulfur analysis was determined by drying separate 1 g samples in a nitrogen atmosphere at 107 °C for 1 h, to a constant weight. The weight of the moisture samples began to stabilize after approximately 40 min. The average moisture measured at NIST for SRM 2775 was 0.66 %, with individual determinations ranging from 0.60 % to 0.74 %.

SRM 2775 moisture measurements obtained by ASTM Subcommittee D05.15 interlaboratory testing ranged from 0.53 % to 0.67 %, when determined in a nitrogen atmosphere, and from 0.54 % to 0.84 % when determined in an air atmosphere.

## PREPARATION, HOMOGENEITY TESTING, AND ANALYSIS

**Preparation:** Foundry coke was collected and crushed to a nominal 2.36 mm (8 mesh) particle size. The crushed coke was subsequently pulverized with ceramic plates until the entire lot passed a 250 µm (60 mesh) sieve. Next, the material was divided by the spinning riffle technique into two portions. One portion was stored in bulk under an argon atmosphere. The other portion was further divided by the spinning riffle technique and bottled under an argon atmosphere.

**Homogeneity Testing:** Twelve bottles from the lot were selected for homogeneity testing. Samples from each bottle were analyzed by X-ray fluorescence for sulfur, strontium, iron, titanium, barium, silicon, calcium, potassium, and aluminum. With the exception of barium, no significant bottle-to-bottle differences were found for any of the elements tested. For barium, a small component of bottle-to-bottle variability was detected. The standard deviation corresponding to this component of variation is estimated to be less than 0.84 % relative, based on a 95 % confidence interval for the standard deviation.

**Reference Values and Uncertainties:** The reference values provided for ash and volatile matter are based on results from interlaboratory testing done in cooperation with ASTM Subcommittee D05.15 with 12 participating laboratories. The uncertainties in Table 2 are given as expanded uncertainties, as described in the NIST ISO Guide [2]. The uncertainties for ash and volatile content were calculated as 95 % confidence intervals, and represent the combined effects of between-laboratories and within-laboratories components of uncertainty. The percent volatile matter obtained by interlaboratory testing was confirmed by measurements made with platinum crucibles under tightly controlled conditions according to ASTM D 3175-89a [5]. Cooperating analysts and laboratories are listed following the table.

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<sup>1</sup>Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The reference values for C, H, and N are based on measurements performed according to a NIST experimental plan by the LECO<sup>1</sup> Corporation (St. Joseph, MI), using ASTM D5373, Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke [7]. The uncertainty in the reference values for carbon, hydrogen, and nitrogen are expressed as an expanded uncertainty,  $U = ku_c$ , calculated according to the methods in the ISO Guide [2] for a prediction interval. The quantity  $u_c$  represents, at the level of one standard deviation, the combined uncertainty due to the potential effects of random measurement errors in the assessment of carbon, hydrogen, and nitrogen and between bottle variance. The quantity  $k = 2$  is the coverage factor used to specify a confidence level of 95 %.

Table 2. Reference Values (Dry Basis)

|                       |                  |
|-----------------------|------------------|
| Ash [3,4]             | 5.77 % ± 0.05 %  |
| Volatile Matter [4,5] | 1.31 % ± 0.20 %  |
| Carbon [7]            | 91.34 % ± 0.49 % |
| Hydrogen [7]          | 0.41 % ± 0.06 %  |
| Nitrogen [7]          | 1.16 % ± 0.05 %  |

#### Cooperating Analysts and Laboratories:

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 E.B. McClain; Koppers Industries, Inc., Dolomite, AL  
 D. Lowenhaupt; Consol Inc., Library, PA  
 L. Janke; Canmet, Ottawa, Ontario, Canada  
 T. Hopkins; New Boston Coke Corp., New Boston, OH  
 D. Chmielewski; Armco Research and Technology, Middletown, OH  
 G. Canoles; Sloss Industries Coke Plant, Birmingham, AL

#### REFERENCES

- [1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; 1995 Ed., U.S. Government Printing Office: Washington, DC (1995); available at <http://physics.nist.gov/Pubs/>.
- [2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.
- [3] ASTM D 3174-93, *Test Method for Ash in the Analysis Sample of Coal and Coke from Coal*; Vol. 05.05, ASTM Book of Standards, West Conshohocken, PA.
- [4] ASTM D 5142-90, *Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures*; Vol. 05.05 ASTM Book of Standards, West Conshohocken, PA.
- [5] ASTM D 3175-89a, *Test Method for Volatile Matter in the Analysis Sample to Coal and Coke*; Vol. 05.05 ASTM Book of Standards, West Conshohocken, PA.
- [6] ASTM D 3172-89, *Standard Practice for Proximate Analysis of Coal and Coke*; Vol. 05.05 ASTM Book of Standards, West Conshohocken, PA.
- [7] ASTM D 5373-93, *Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke*; Vol. 05.05 ASTM Book of Standards, West Conshohocken, PA.

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| <b>Certificate Revision History:</b> 10 November 2005 (This revision adds reference values for C, H, and N); 01 May 1997 (Original certificate date). |
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*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*